

Biodiesel Synthesis from Used Cooking Oil Using Red Mud as Heterogeneous Catalyst

Arif Hidayat^{1,a*}, Galih Kholifatu Roziq^{1,b}, Faiz Muhammad^{1,c},
Winarto Kurniawan^{2,d} and Hirofumi Hinode^{2,e}

¹Chemical Engineering Department, Universitas Islam Indonesia, Indonesia

²Global Engineering for Development, Environment and Society, Transdisciplinary Science and Engineering, Tokyo Institute of Technology, Japan

^{a*}arif.hidayat@uui.ac.id, ^b15521098@students.uui.ac.id, ^c15521120@students.uui.ac.id,
^dkurniawan.w.ab@m.titech.ac.jp, ^ehinode.h.aa@m.titech.ac.jp

Keywords: methyl ester, used cooking oil, red mud, biodiesel, heterogeneous catalyst.

Abstract. The problem associated with biodiesel production is economic feasibility. The biodiesel cost will reduce when the low cost feedstock was used as feedstock. Used Cooking Oil (UCO) is a promising candidate as raw material for biodiesel synthesis. In this study, the investigation of biodiesel synthesis from UCO was studied using red mud as heterogeneous catalysts. The catalyst was prepared by impregnating Potassium metals on red mud. The catalyst physico-characteristics were determined using Nitrogen gas adsorption, FT-IR, XRD, and XRF. The catalyst was tested to synthesize biodiesel from UCO. The reaction temperatures, methanol to oil mass ratio, and amount of catalyst were varied to examine their effects on biodiesel synthesis. The optimum reaction conditions were obtained at 60°C of reaction temperature, 10:1 of methanol to oil mass ratio, and 10% of catalyst amount. The highest biodiesel yield of 94.4% was obtained.

Introduction

Several factors that lead to the search for alternative energy sources are increasing energy demand, global warming due to greenhouse gas emissions, environmental pollution, and reduced fossil fuels supply [2]. Biodiesel has been proven to replace diesel fuel. Biodiesel contains of monoalkyl fatty acid esters with long chain of hydrocarbons. Biodiesel has several benefits such as emit less pollutant substances, biodegradable, generates from renewable sources and reduce greenhouse gases production [14]. Conventionally, transesterification of triglycerides and esterification of free fatty acids were carried out to yield biodiesel using assistance of acid or base catalysis. However, the problems encountered with the biodiesel synthesis are the expensive of feedstocks cost and the emergence of competition between energy and food supply [10]. Currently, more than 95% of biodiesel synthesis has been using various type of edible oil, such as canola, palm, corn, sunflower and soybean [4]. Nevertheless, the use of edible oil lead to the uneconomically viable of biodiesel price compared to petrodiesel. Reducing the feedstocks cost can be achieved by using non-edible oils and waste cooking oils as raw materials. The non-edible oils have low price and easy cultivating in land-poor. Energy crops such as jatropha [15], mahua [13], karanja [11], Ceiba pentandra [16], Calophyllum inophyllum [3], and rubber seed [8] represent some of non-edible oils plant. Used cooking oils (UCO's) are oils or animal fats that have been used for cooking or frying in the food processing industry, restaurants, and households. UCO's have a low-cost which available abundantly and sustainable. Utilization of UCO's as a feedstock will increase the economic viability in term of reducing of biodiesel price.

In the last decades, many researchers have been studied different heterogeneous catalysts for biodiesel synthesis [1, 5-7, 9, 12]. Many advantages was gained such as easily separated from the product mixture, produce less wash water and can be reused when applying the heterogeneous catalysts for biodiesel synthesis compared to the homogeneous catalysts. From the literature study, it has been summarized that most of the heterogeneous catalysts preparation have time consuming preparation, complex synthesis routes, and expensive. To address these drawbacks, a new type of

heterogeneous catalyst from tailing residue needs to develop. Red mud is a residue from extraction process of bauxite mineral to produce alumina in the Bayer process. The pH of red mud is high with in the range between 10–13 due to the usage of sodium hydroxide during alumina extraction from bauxite. Red mud contains several metals oxide such as Al_2O_3 , SiO_2 , Fe_2O_3 , TiO_2 and more which is regarded as a hazardous waste. Red mud which has high alkalinity is potential to utilize as a heterogeneous basic catalyst for biodiesel production. The red mud utilization as a heterogeneous catalyst will emerge several benefits i.e. low cost, environmentally benign, and exhibits high activity during biodiesel synthesis.

This study focused on the biodiesel synthesis from UCO using red mud as catalyst. The incipient wet impregnation procedure was applied to synthesize the catalysts. The catalyst physico-characteristics were determined using Nitrogen gas adsorption, FT-IR, XRD, and XRF. The investigation of several operating conditions in term of the reaction temperature (40 – 60°C), the methanol to UCO mass ratio (1:2 – 2:1), and the amount of catalyst to oil (1 – 10% wt. of UCO) were varied to obtain the optimum reaction conditions.

Experimental

Catalyst Preparation. The Ca/RM catalysts were synthesized by the incipient wet impregnation using metal salts solution of Calcium Nitrate (CaNO_3). Then, the red mud as catalyst support was mixed with a Calcium Nitrate solution and stirred vigorously while the temperature was heated up to dry the mixture. When the slurry of mixture was formed, the mixture was then calcined at 600 °C in a furnace for 2 hours. The textural characteristics of red mud and Ca/RM catalysts were analyzed using the Nitrogen adsorption isotherm at 77 K using Quantachrome ASAP 2010 instrument. The structure and crystallinity of materials were identified by using a Rigaku Multiflex X-ray diffractometer, using radiation of $\text{Cu K}\alpha$, the high voltage source of 40 kV and 20 mA. Datas were recorded with scanning angle between 5 to 80° (2θ) at a scanning rate of 1°/min. The X-Ray Fluorescence (XRF) spectrophotometer analysis was applied to determine elemental composition of the materials. A series of Hammett indicators i.e. phenolphthalein ($H_- = 8.2$), 2,4-dinitroaniline ($H_- = 15$) and 4-nitroaniline ($H_- = 18.4$) was used to determine the basic strength of Ca/RM catalyst.

Results and Discussion

Catalyst Characterization. The structure and crystallinity of the raw red mud and Ca/RM catalysts was determined by applying XRD analysis. The results of diffraction pattern peaks analysis from XRD measurements were depicted in Fig. 1. As illustrated in Fig. 1, the XRD diffractogram exhibits several metal oxides on red mud such as Fe_2O_3 (hematite), Al_2O_3 (boehmite), and TiO_2 (anatase titania) as major mineral phases. Meanwhile, on the Ca/RM catalysts the characteristics peaks of CaO were shown at $2\theta = 19, 23, 29, 32$ and 34° . In addition, small peak of CaO was displayed at $2\theta = 31^\circ$. It is noteworthy that, the incorporated of potassium metals on red mud was succeed by the presence of the presence of characteristics peaks CaO of well as of on XRD Diffractogram. The findings in line with XRF results, in which potassium content in the Ca/RM catalyst increased after impregnation. The surface area was determined by N_2 adsorption isotherm analysis. The surface area of Ca/RM catalysts was $20.2 \text{ m}^2/\text{g}$, while that of raw red mud was $24.8 \text{ m}^2/\text{g}$. The reducing of surface area was attributed to the successful incorporating of potassium oxide to pores as confirmed in the results from XRD analysis. The strong base of Ca/RM catalysts were analyzed using Hammet indicators. The results showed that the phenolphthalein colour ($H_- = 8.2$) was changed from colourless to pink as well as the 2,4-dinitroaniline colour ($H_- = 15$) from yellow to mauve. However, the 4-nitroaniline colour ($H_- = 18.4$) has not changed. It concluded that, the Ca/RM catalyst's is considered as a strong base catalyst due to the designated of basic strength was in the between $15 < H_- < 18.4$.

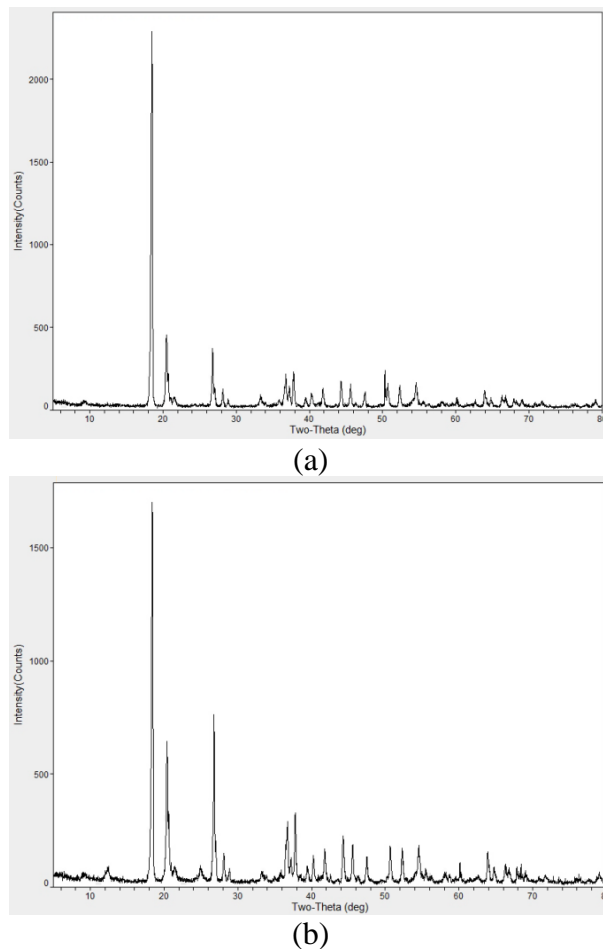


Fig. 1. XRD measurements: (a) raw red mud; and (b) Ca/RM catalysts

Catalyst Activity. The transesterification reaction is reversible, thus it needs to employ a methanol excess to keep the reaction in the forward direction. The effects of methanol to KSO mass ratio on the biodiesel yield in the transesterification reaction of UCO were showed in Fig. 2. As illustrated in Fig. 2, the yield of biodiesel was 74% when methanol to UCO mass ratio was applied at the value of 1:1. Increasing of methanol to UCO mass ratio to 2:1 increased the biodiesel yield to 89%. The further increasing of methanol to UCO mass ratio from 3:1 to 4:1 did not significantly enhance the biodiesel yield. Therefore, the maximum biodiesel yield was achieved at a 2:1 methanol to UCO mass ratio.

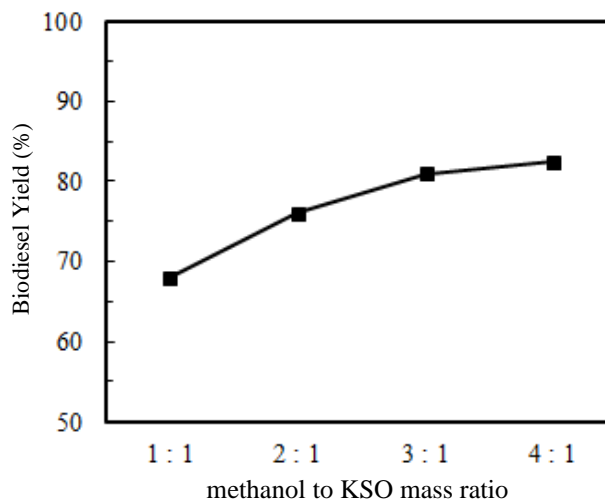


Fig. 2. Influence of Methanol to UCO Mass Ratio on Biodiesel Yield

The one of important parameters that affects the biodiesel yield is catalyst amount. The value of catalyst amount was varied from 1 to 10 wt. % UCO. As presented in Fig. 3, the biodiesel yield was enhanced when catalyst amount from increased from 2.5 to 5 wt. % UCO. The maximum biodiesel yield of 86% was attained at a catalyst amount to 10 wt. % UCO. The increasing of catalyst amount lead to more total number of available active sites resulted in faster reaction rate to achieve reaction equilibrium and generated more FAME.

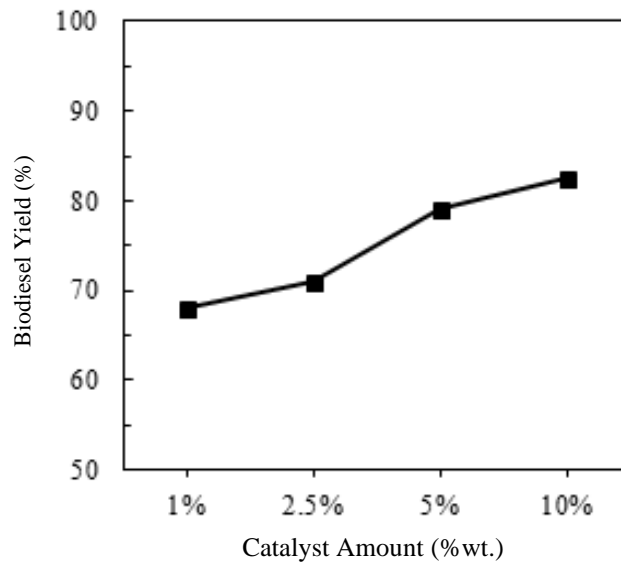


Fig. 3. Influence of Catalyst Amount on Biodiesel Yield

The variations in reaction temperatures are applied to investigate their influence of on the biodiesel yield as shown in Fig. 4. The different reaction temperatures were applied from 40 to 60 °C min to obtain the optimum reaction temperature for reaction. The increasing of reaction temperature from 40 to 50 °C resulted the enhancing of biodiesel yield from 76 to 83%. It was observed that at a reaction temperature of 60 °C, the biodiesel yield reached maximum value at 88%. At high temperature, the limitation between mixture of reactant and catalyst would reduce and the reactant molecules also gained more kinetic energy. It caused an accelerating of reaction rate between reactants that resulted to produce more FAME.

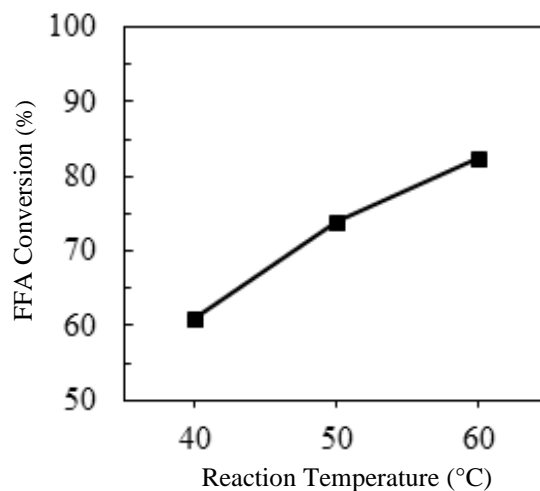


Fig. 4. Influence of Reaction Temperature on Biodiesel Yield

Conclusions

Red mud which a residue from bauxite processing was employed as catalyst support for biodiesel production from Used Cooking Oil. The catalyst was prepared by impregnating the red mud with Potassium metal. The optimum reaction conditions were obtained at 60°C of reaction temperature, 10:1 of methanol to oil mass ratio, and 10% of catalyst amount. The highest biodiesel yield of 83% was obtained.

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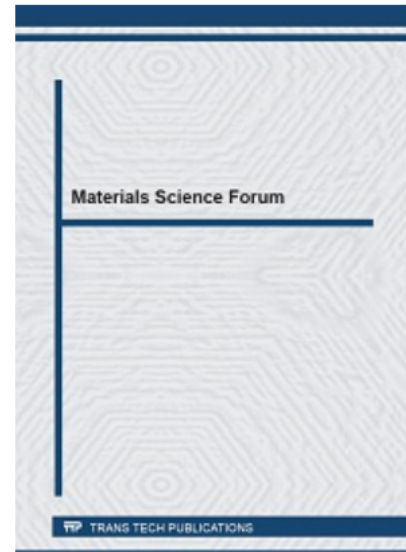
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Table of Contents

Preface

Chapter 1: Metallurgical Technologies, Properties of Steels and Alloys

Microstructure and Impact Toughness of Flux-Cored Arc Welded SM570-TMC Steel at Low and High Heat Input H. Oktadinata, W. Winarto and E.S. Siradj	3
Investigation of the Intermetallic Formation on Wet Underwater Welding of AISI 4012 Steel A.D. Anggono, Suwantri, W.A. Siswanto and J. Sedyono	10
Effect of Magnesium on the Strength, Stiffness and Toughness of Nodular Cast Iron A.S. Darmawan, P.I. Purboputro, A. Yulianto, A.D. Anggono, Wijianto, Masyrukan, R.D. Setiawan and N.D. Kartika	17
Effect of Ti Contents on the Microstructure and Mechanical Properties of NiAlTi System D. Wicaksono, X.M. Zhu, M.S. Mustapa, S. Yulianto, A.Y. Nasution and T.W.B. Riyadi	24
Defect Investigation of Sand Casted Aluminum Cooling Fan D. Prihtiantoro, A.D. Anggono and W.A. Siswanto	30
Microstructure and Hardness of Gray Cast Iron as a Product of Solidification in Permanent Mold A. Yulianto, R. Soenoko, W. Suprpto, A. Sonief, A.S. Darmawan and M.D. Setiawan	37
Structure and Properties of NiAlTi Systems Formed by Combustion Synthesis T.W.B. Riyadi	44

Chapter 2: Characterization and Testing of Materials

HAp Coated Hip Prosthesis Contact Pressure Prediction Using FEM Analysis M. Nagentrau, A.L. Mohd Tobi, S. Jamian and Y. Otsuka	53
Effect of the Fine Recycled Aggregates on the Dynamic Compressive Behavior of Recycled Mortar S. Ismail, M.A. Abd Hamid and Z. Yaacob	62
Assessment of the Stress-Strain State of a Tube Sheet of the Heat Exchanger at Rotary Friction Welding Application A.S. Tokarev, D. Karetnikov, R.G. Rizvanov, A.M. Fаnгуshin and M.Z. Zariрov	70
Characterisation of Electrode Drying Effect on the Tungsten Carbide Hardfacing Microstructure M. Nagentrau, A.L. Mohd Tobi, S. Jamian and M. Sambu	77
Fatigue Behavior Improvement of A356 Aluminum Alloy of Motorcycle Cast Wheel Produced by High Speed Centrifugal Casting Based on T6 Heat Treatment and Artificial Aging P.T. Iswanto, Akhyar, A. Janata, L.M. Mauludin and H.M. Sadida	86
Electrochemical Performance of Barium Strontium Cobalt Ferrite -Samarium Doped Ceria- Argentum for Low Temperature Solid Oxide Fuel Cell U.A. Yusop, T.K. Huai, H.A. Rahman, N.A. Baharuddin and J. Raharjo	94
Resistance to Chloride Penetration of Recycled Aggregate Concrete Modified Using Treated Coarse Recycled Concrete Aggregate and Fibres S. Ismail and M. Ramli	101

Chapter 3: Technologies of Biomass Processing

Catalytic Pyrolysis of Palm Empty Fruit Bunch over Activated Natural Dolomite Catalyst: Product Distribution and Product Analysis A. Hidayat, M.A. Adnan and A. Chafidz	111
---	-----

CaO/Natural Dolomite as a Heterogeneous Catalyst for Biodiesel Production B. Sutrisno, A.D. Nafiah, I.S. Fauziah, W. Kurniawan, H. Hinode and A. Hidayat	117
Investigating the Potential Use of Cassava Leaf Extract as a Natural Coloring Substance for Fabrics S. Rusdi, M.Y. Zakaria, R.N.F. Aditya and A. Chafidz	123
Investigating the Potential Use of Papaya Leaf Extract as Natural Dyes in the Textile Industry S. Rusdi, H.F. Maulana, N.L. Samudro and A. Chafidz	129
Synthesis of Grafted Cationic Starch with DMDAAC Using Ammonium Persulfate/Carbamide Initiation System H. Tang, P.Y. Zhang, T.X. Li and Y. Ma	135
Biodiesel Synthesis from Used Cooking Oil Using Red Mud as Heterogeneous Catalyst A. Hidayat, G.K. Roziq, F. Muhammad, W. Kurniawan and H. Hinode	144
Effect of HCl-Alcoholic Treatment on the Modification of Jackfruit (<i>Artocarpus heterophyllus</i> Lam) Seed Starch T.H.T. Le, H.T. Nguyen, V.K. Nguyen, T.L. Nguyen and T.T. Nguyen	150

Chapter 4: Technologies of Chemical Production and Wastewater Treatment

Characterization of Hydroxyapatite Synthesized from Calcium Hydroxide and Phosphoric Acid as Adsorbents of Lead in Wastewater H.T. Nguyen and P.T. Dang	159
Research and Application of a New Demulsifier for the Processing of Produced Liquid in Changing Gasfield S.J. Chen, F. Tang, W. Tian, Q.n. LIU and G. Chen	166
Utilization of Modified Zeolite Materials as Chromium Cation Exchanger for Treatment of Liquid Waste from Electroplating Industries Z. Salimin, M. Susianto, B. Batara and A. Chafidz	172
Chemical Treatment of Liquid Waste Generated from Leather Tannery Industry by Using Alum as Coagulant Material Z. Salimin, F.W. Satiyoaji, D.A. Prasetya and A. Chafidz	178
Synthesis of Hydrophobically Associating Polymers and the Application as Oil-Displacing Agent R.J. Zhang, J.L. Zhao, X.K. Wang, Z.P. Zhou and G. Chen	185
Corrosion Rate Analysis of API 5L Gr B Steel Pipe in Acetic Acid Contained Crude Oil Treatment System by Using Amine Base Organik Corrosion Inhibitor A. Swandito and V. Malau	191

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Prof. Jai Sung Lee

Hanyang University, Department of Metallurgy and Materials Science; 55 Daehak-no, Sangnok-gu, Ansan, Korea, South, 426-791;

Prof. Eric J. Mittemeijer

Max Planck Institute for Intelligent Systems; Heisenbergstrasse 3, Stuttgart, 70569, Germany;

Prof. Stephen J. Pearton

University of Florida, Department of Materials Science and Engineering; Gainesville, USA, 32611-6400;

Prof. Vassilis Pontikis

Commissariat à l'Energie Atomique et les Energies Alternatives (CEA), CEA-Saclay; Bdg. 524, Gif-sur-Yvette, 91191, France;

Prof. András Roósz

Hungarian Academy of Sciences, Miskolc University (HAS-MU); Miskolc-Egyetemváros, 3515, Hungary;

Prof. David N. Seidman

Northwestern University, Department Materials Science and Engineering; Cook Hall, 2220 Campus Drive, Evanston, USA, 60208;

Dr. Ching Hua Su

NASA/Marshall Space Flight Center, EM31 NASA/Marshall Space Flight Center; Huntsville, USA, 35812;

Prof. David Tomanek

Michigan State University, Physics and Astronomy Department; 567 Wilson Road, East Lansing, USA, MI 48824-6455;

Prof. A.S. Wronski

University of Bradford, School of Engineering, Design and Technology; West Yorkshire, Bradford, United Kingdom, BD7 1DP;

Emeritus Prof. David J. Young

University of New South Wales, School of Materials Science and Engineering; Sydney, Australia, NSW 2052;

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Universiti Teknologi Brunei (UTB)
Brunei



Prof. Dr. Muhaimin Ismoen
Computational nanomaterials, Computational fluid dynamics, Numerical methods
Universiti Teknologi Brunei (UTB)
Brunei



Mr. Agung Setyo Darmawan

Biomaterial
Universitas Muhammadiyah Surakarta
Indonesia





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Finite element analysis, Simulation, Design and Modeling, Manufacturing

Universitas Muhammadiyah Surakarta (UMS)

Indonesia



Mr. Agus Mujianto

Mechanical Engineering Materials

Universitas Muhammadiyah Kalimantan Timur (UMKT)

Indonesia



Mrs. Anis Siti Nurrohkayati

Industrial Engineering, Manufacture System

Universitas Muhammadiyah Kalimantan Timur (UMKT)

Indonesia



Mr. Ardiansyah

Materials

Universitas Muhammadiyah Kalimantan Timur (UMKT)

Indonesia



Mr. Binyamin

Solid Mechanics, Materials, Finite Element, Energy

Universitas Muhammadiyah Kalimantan Timur (UMKT)

Indonesia



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Advanced materials, manufacturing

Universitas Mercu Buana (UMB)

Indonesia



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Renewable Energy

Universitas Muhammadiyah Kalimantan Timur (UMKT)

Indonesia



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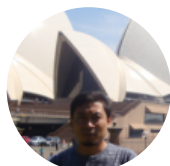
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Universiti Tun Hussein Onn Malaysia (UTHM)

Malaysia



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Materials Manufacturing, Advanced Machining, Machine Design

Universiti Tun Hussein Onn Malaysia (UTHM)

Malaysia



Mr. Nagentrau Muniandy

Mechanics of material, Finite element method, Hardfacing

KDU University College

Malaysia



Dr. Sharifah Adzila Binti Syed Abu Bakar

Advanced materials, Biocompatible Materials, Biomaterials

Universiti Tun Hussein Onn Malaysia (UTHM)

Malaysia



Ts. Dr. Zakiah Binti Kamdi

Material sciences, Advanced materials

Universiti Tun Hussein Onn Malaysia (UTHM)

Malaysia



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Materials Engineering, Inorganic Chemical Engineering, Environmental Engineering

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